

FACILITY FORM 602

N64-27804

(ACCESSION NUMBER)

39

(PAGES)

CR-58053

(NASA CR OR TMX OR AD NUMBER)

(THRU)

1

(CODE)

15

(CATEGORY)

OTS PRICE

XEROX

\$ 3.60 ph.

MICROFILM

\$

Final Report to
Jet Propulsion Laboratory

Spherical Rotors for Cryogenic Gyroscopes

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March 15, 1962

**This work was performed for the Jet Propulsion Laboratory,
California Institute of Technology, sponsored by the
National Aeronautics and Space Administration under
Contract NAS7-100.**

Nuclear Metals, Inc.
Concord, Massachusetts

Contract No. 950032
January 3, 1961 through ~~January 3, 1962~~

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ABSTRACT

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The development and fabrication of special rotors for cryogenic gyroscopes was undertaken. Lucalox (alumina, General Electric) and Homosil (fused quartz, Englehard Industries) spheres 4.00 cm in diameter were used as rotor substrates. The spheres were coated with niobium vapor-deposited from NbCl_5 using hydrogen as a carrier gas. The spherical substrate was simultaneously suspended, rotated, and heated to about 1000°C in a hot-gas bearing; the gas was argon issuing from a plasma torch. Bright, metallic coatings several tenths of a micron thick were produced on an equatorial band around the spheres. The coatings were very adherent, even after thermal cycling, and had a low electrical resistance at 300°K . The best coating, on a Lucalox substrate, was at least partially superconducting. A photoresist-anodization technique was demonstrated for accurately placing marks on the niobium for optical readout.

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I. INTRODUCTION

Space navigation and advanced weapons systems require guidance systems of ever-increasing stability and accuracy. Gyroscopes are usually the core of such guidance systems, and finely balanced gyrorotors, in nearly frictionless suspensions, must be achieved.

The objective of this program was to fabricate niobium-coated spheres for use as cryogenic rotors. The design specifications for these rotors are given in JPL Specification No. 15008B (see Appendix A). For convenience, the program was divided into the following parts: (1) procurement of substrate spheres with a high degree of isotropy, uniformity and purity, and surface grinding these to required sphericity; (2) coating of the substrate spheres with a thin, uniform layer of niobium metal by vapor deposition and measuring some of the properties of the coated spheres; and (3) providing the coated spheres with an optical pattern to aid in determining rotor orientation.

The prior studies made in order to achieve the objective with minimum cost and time are summarized in Appendix B. These also justify the particular choice of materials and processes for this program.

II. PROCUREMENT AND PROCESSING OF SUBSTRATE MATERIAL

At the outset of this program three substrate materials were considered: aluminum oxide (Al_2O_3), fused quartz, and beryllium. Beryllium was dropped early in the program because spheres of metal are more difficult than spheres of ceramic to grind to the required sphericity tolerances. Orders were placed for 10 Lucalox (General Electric Co.) high density Al_2O_3 spheres (1-3/4 in. diameter) and two "Homosil Grade" fused quartz blanks (Amersil Quartz Division, Englehard Industries). The original order for 10 Homosil blanks was reduced to two because of the relatively high cost and delayed delivery.

The Lucalox spheres were examined by X-ray radiography and no pores or inclusions were found. Figure 1 is a radiograph of the spheres as received from the General Electric Co. The slight non-uniformities in the radiograph are attributed to the lack of sphericity in the blanks.

Since this examination revealed no flaws, the 10 Lucalox spheres were sent to Industrial Tectonics, Inc., Ann Arbor, Michigan, for precision grinding to finished size.

Cursory inspection of the finished Lucalox spheres indicated that they were of high quality. However, microscopic examination (30X) revealed numerous small pits, some containing a black substance. These pits were approximately 0.004 in. across and up to 0.004 in. deep. Industrial Tectonics has furnished the following information concerning the spheres:

1. The specific diameter of the balls is 1.5728 in.
2. The balls are out-of-round between 0.0001 and 0.0002 in. At one stage the sphericity was within 0.000050 in., but because of the surface condition it was necessary to run the process further and the balls became less spherical. It is felt that additional experience with this material could result in attainment of a sphericity of 0.000010 in.
3. Although the black, hard, metallic spots on the Lucalox blanks, as received from General Electric, disappear as the diameter is reduced, they give rise to the pits that persist during grinding. A similar phenomenon has occurred on another batch of smaller Lucalox balls being processed for another company. This phenomenon is regarded as very peculiar since the grinding process normally causes a defect of this type to disappear rather than result in a pit that is carried down to the finished diameter.

The Homosil quartz blanks were examined visually, when received, after placing them in carbon tetrachloride. This liquid has excellent optical clarity, freedom from bubbles, and a close match in refractive index (1.463 at 589 m μ , the sodium D lines) to fused quartz (1.458 at 589 m μ). The blanks were found to be bubble- and defect-free, and they showed no internal strains when inspected under polarized light.

Because it is uneconomical and not technically desirable to process only two spheres at a time at Industrial Tectonics, these blanks were sent

to the A. D. Jones Optical Company, Cambridge, Massachusetts for grinding and polishing to final size. Visual examination of the surface of the polished spheres at 30X revealed no pits or scratches. Based upon the relative equipment and experience of these companies, it was not expected that the quartz spheres would have as high a degree of sphericity as the Lucalox ones. The A. D. Jones Optical Company has stated that the two quartz spheres were out-of-round by about 0.0001 in.; hence, the quartz was comparable to the Lucalox in sphericity. Although the spheres of both materials did not meet the desired specifications, they were adequate for study of their suitability as substrates for niobium coatings.

III. COATING OF SUBSTRATE SPHERES WITH NIOBIUM

A. Apparatus and Procedures

Preliminary studies indicated that the most favorable solution to the coating problem is vapor deposition from gaseous niobium pentachloride (NbCl_5) onto a sphere floating in a hot gas bearing. Apparatus to accomplish this has been constructed and is illustrated in Figs. 2, 3, and 4 and described below.

A niobium-coated, precision-ground, graphite bearing is positioned over a plasma torch. This assembly is encased in a small, stainless steel, water-cooled chamber attached via an O-ring seal to the plasma-torch housing. A glass still with a heating mantle is connected by a heated glass tube to the chamber. Opposite this inlet is a vacuum outlet that leads through a series of glass collecting-bottles (filtering flasks) and a mercury manostat to a vacuum pump. High purity argon, helium and hydrogen gases are brought to the system from high pressure tanks through a gas clean-up train consisting of standard Englehard "Deoxo" units followed by drying chambers using Linde A molecular sieves.

In operation, a sphere is placed in the bearing and the chamber is sealed and evacuated. The system is flushed with argon mixed with a trace of hydrogen. The hydrogen combines in the Deoxo unit with any residual oxygen that is in the gas stream, and the resulting H_2O is removed in the

molecular sieve. This gas is then allowed to continue flowing through the plasma torch. The argon now serves as both the plasma gas and the gas-bearing suspension medium. With the gas flow and vacuum lines properly adjusted, the current generator is turned on, the plasma arc is started, and the current slowly increased as the sphere is brought slowly to about 1000°C . Meanwhile, commercially pure (see Table I) NbCl_5 , previously loaded into the still, is heated to the volatilization point and conducted to the deposition chamber using hydrogen as a carrier gas. This gas mixture is directed at the top of the sphere in the bearing where the niobium is vapor deposited. The by-products of the reaction are caught in the collecting bottles or removed by the vacuum pump. After sufficient time for plating has passed, the plasma torch is reduced to the lowest operating temperature possible. Finally, everything is turned off except the water and plasma gas, which are continued until the sphere is cooled. A typical run on a Lucalox sphere is described in Table II.

In the original apparatus a steel gas bearing was used. However, a graphite bearing was later substituted for two reasons: (1) as various designs and sizes of bearings became necessary (each bearing must be very close to the size of the supported sphere for smooth operation) it became apparent that machining costs would be minimized with graphite; (2) the graphite bearing heats more uniformly to a higher temperature than the steel, and hence promotes a more uniform heating of the sphere. A few runs with graphite gas bearings resulted in black, high-resistance niobium coatings. It was suspected that an interaction of niobium with carbon had occurred. Hence, all the later runs were made with graphite gas bearings that were coated with niobium on all surfaces (exterior and interior, facing the sphere and facing the plasma torch). This coating was carried out by the same process of vapor deposition from NbCl_5 .

Four other problems were encountered in the development of this apparatus. Three of these problems concerned the sphere, and were: bobbling

of the sphere in the bearing, narrow band heating, and inability to vary the axis of rotation. These problems were mitigated by the following innovations.

Bobbling of the spinning sphere was stopped by isolating the plasma entrance chamber from the support annulus (the gas bearing itself) with small holes. These holes effectively decoupled the instabilities of flame and sphere from each other. Figure 4 is a sketch of this portion of the equipment. Bobbling may also be eliminated by increasing the pressure in the decomposition chamber from about 125 mm Hg to about 380 mm Hg.

Narrow band heating occurs when energy transfer from plasma to sphere takes place over a localized region. When the sphere is spinning, this mode of heating results in a great-circle band of increased temperature with the rest of the sphere soaking up the heat slowly by conduction through the sphere material. Stressed spheres, prolonged runs, and extremely non-uniform coatings have resulted from such a condition. This problem was overcome by coupling the plasma to the sphere through many off-center holes rather than a few holes near the axis (see Fig. 4).

The axis of rotation of the spheres should be varied to promote a uniform coating by averaging out any shadowing effects. This problem was not solved during the course of this program. Changes of rotation of axis can be achieved by using a fairly precise symmetrical bearing contour (± 0.0001 inch) and a relatively slow spin rate. However, the maintenance of slow spin rates while the sphere changes rotation axis (tumbles at random) is difficult. Because of small geometrical irregularities or because of small instabilities, the sphere will commence rotating about a certain axis. This axis then becomes preferred because a Bernoulli effect within the gas bearing pushes the sphere towards one side of the center line and the annulus then becomes asymmetrical. The sphere then increases in angular velocity about this axis until it becomes a gyro. At this point, the effect of steering jets is merely to introduce a precession, unless very large torques are available. The steering jets originally put into the equipment were of insufficient maneuverability and torque to regulate the sphere velocity and axis of rotation, and the jets were subsequently eliminated. Consequently, the final coatings consisted of broad bands centered

on the equators of the spinning spheres; i.e., no attempt was made to stop the natural tendency of the spheres to become gyros, emphasis being given, instead, to the production of good coatings without regard to their uniformity. The solution to this uniformity problem is mechanical in nature, and this problem does not pose any fundamental, insurmountable difficulty to the achievement of uniform coatings.

The fourth problem concerned the distillation of NbCl_5 . A first attempt at multiple distillation proved futile because of temperature effects on teflon stopcocks. These stopcocks were chosen because they were greaseless and it was thought they could withstand the temperatures (about 480°F) necessary to distill NbCl_5 . However, at distillation temperature they deformed and destroyed the vacuum seal.

A solution was found to this problem of performing multiple distillation without stopcocks or intermediate sealing and breaking of glass. This solution employs a multi-stage still of continuously connected glass (no stopcocks), the transfer sections between the stages being necked down. When isolating each stage, only the transfer section in use is heated, the others are kept cold. NbCl_5 plugs will form in the constricted, cold, sections. These plugs will hold a pressure of at least 3×10^{-2} mm of Hg and therefore act as stopcocks. Instead of this scheme, only a single distillation was carried out in practice because of the expense involved in multiple distillation and because a fairly high purity NbCl_5 can be obtained using commercially available material (Table I) and a single distillation. All the NbCl_5 used in this program was Stauffer Chemical Corp. material. It was noted that a white cake was invariably left as a residue in the distillation flask after all vaporization of yellow NbCl_5 had ceased. The composition of this residue was not determined, but it is suspected to be another niobium chloride not volatile at these temperatures.

B. Coating Results

Many of the coating trials were abortive for reasons other than the occasional malfunctions of the equipment. Several trials at the beginning of the program resulted in black coatings of high electrical resistance. However, the use of niobium-coated gas bearings appeared to eliminate this

difficulty. Some coatings that might otherwise have been satisfactory were damaged by contact with the gas bearing while the spheres were rapidly spinning.

In general, those coating runs that were most successful on polished spheres yielded deposits that appeared highly reflective, bright and metallic. These deposits were of low resistance at 300°K (several ohms or less as measured on an ohmmeter with probes at the ends of a diameter) and were tightly adherent to the substrate. Thermal cycling between about 300°K and 77°K produced no changes in the character of the deposited film as observed visually, microscopically (up to 30X), and by electrical resistance measurements. These coatings were a few tenths of a micron thick as determined by weight gain of the spheres.

All of the coatings were deposited as a broad equatorial band. After the difficulties were experienced with the gyro effect of the sphere in the gas bearing, efforts to improve coating uniformity were set aside in favor of producing the equatorial coatings for evaluation of their superconducting properties.

It was noted, especially for the quartz spheres, that the coating did not always build up uniformly, but seemed to spread over the surface by nucleation and growth.

Removal of the coatings by chemical etching from both quartz and Lucalox spheres showed that the quartz was attacked during the deposition. In those regions that were covered with a niobium deposit the quartz surface had a mottled, or etched, appearance whereas the rest of the surface retained its original polish. This phenomenon was not observed with the Lucalox. It is not known whether the quartz interacted directly with the niobium or whether the hydrogen or chlorine ions participated. In spite of this interaction, the coatings on quartz were bright, metallic, and of low resistance.

The last, and best, coated sphere in this program was of Lucalox equatorially coated with niobium several tenths of a micron thick. This sphere was sent to the Jet Propulsion Laboratory for evaluation of its superconducting properties. It was found to be superconducting, although

the critical field was anomalously low. After several weeks, the coating lost its superconducting nature and appeared to be slightly discolored. No protecting layer was provided on the film since the rate of oxidation of bulk niobium is very low under ambient conditions. However, this particular film was also very thin and the loss of superconductivity may be attributed to reaction with the atmosphere.

IV. OPTICAL PATTERN

The object of this phase was to provide the surface of the niobium-coated spheres with an optical pattern for photocell readout of sphere orientation. The objective was met by using photoresist techniques to develop a latent pattern that permitted selective anodization on the niobium surface to develop the permanent pattern. This phase of the program was carried out under subcontract with CBS Electronics, Lowell, Massachusetts.

At the special request of Jet Propulsion Laboratory, the technique was developed on half-inch solid niobium spheres. Four equatorial marks, 90° apart, and one mark at each pole were requested. Two approaches were taken for achieving the photoresist pattern. One utilized planar masks and the other a cylindrical mask.

In the first approach the sphere was suspended between two planar masks by means of the pressure created by a jig which holds and orients the masks. In this process two exposures, one from each side, were necessary. However, the resolution was very poor in the region about 90° from the mask-sphere contact points. Because both polar and equatorial markings were desired, this approach was abandoned in favor of the second one.

In the second approach a hollow plastic cylindrical jig (Figs. 5 and 6) with machined ends was used. This held a cylindrical mask in contact with the equator of the sphere and two planar masks in contact with the poles. Appropriate funnels for light transmission were machined into the cylinder and its ends. Good definition was thus provided at all regions where patterns were to be applied.

To produce an optical pattern on a sphere it was first coated with a KMER resist (Eastman Kodak Company) solution diluted 30% by volume with trichlorethylene. The coating was applied by dipping the sphere and allowing the excess resist to drain down over the contact area. Then the sphere was turned 180 degrees, dipped and the excess resist allowed to drain over the other contact area to completely cover the sphere. It was then placed in the above jig, called a "ball exposure cage", with the appropriate masks, and exposed to a carbon arc light source. The resist was about 16 inches from the source and exposed for four minutes. The arc source was formed by 1/4-in. copper-coated carbon electrodes operating at 7 amperes. The inside of the cage was blackened to prevent stray light leakage into the cage, which would cause spotty exposure of the resist and consequent spotty anodization of the sphere.

The sphere was next removed from the cage and placed in the anodizing fixture (Fig. 7), care being taken not to mar the previously exposed patterns. It was developed using a standard developer solution (Eastman Kodak Company). Any uncoated areas, as well as the circumference of the contact electrodes, were protected with wax before anodizing.

The anodizing process took place in 1N H_2SO_4 at room temperature for a maximum of 30 seconds at 20 volts yielding a blue interference color. Larger anodizing times resulted in pinholing of the resist and complete anodization of the ball.

Upon completion of this process the remaining KMER resist was removed in a vapor degreaser by exposing the sphere for 30 minutes in trichlorethylene, and then washing in a water stream.

The master planar drawing from which the working flexible positive is made was accurate to ± 0.0001 with respect to pattern configuration and pattern location. The one-half inch spheres, as received from the Jet Propulsion Laboratory, were actually 0.485 in. in diameter whereas the cage was tooled to 0.500 ± 0.001 in. This resulted in an accuracy of ± 0.005 in. for pattern location. With minor modifications in the cage design, and with spheres more closely matched to the cage, pattern location accuracy can be kept to the order ± 0.002 in.

The anodized film is extremely thin, 300-1000 Angstroms, and of high optical contrast. It is very abrasion resistant and withstands the thermal shock of rapid cycling between 77°K and 300°K .

V. CONCLUSIONS

The following conclusions have been drawn from this program:

1. Both fused quartz and Lucalox may ultimately be acceptable as rotor substrates.
 - a. Fused quartz of acceptable quality is available.
 - b. Fused quartz is much easier to inspect throughout its volume than is Lucalox.
 - c. Fused quartz is chemically attached during the vapor deposition whereas Lucalox is not.
 - d. Grinding and polishing of Lucalox spheres appears to require further development to avoid pitting.
2. Vapor deposition of niobium can yield adherent films possessing at least some superconducting characteristics.
 - a. Excellent adherence of films a few tenths of a micron thick was observed on both fused quartz and Lucalox even after thermal cycling between 77°K and 300°K .
 - b. The vapor deposited films, when properly produced, are bright, metallic, and mirror-like.
 - c. These films have a low resistance at 300°K and, at least on Lucalox, may exhibit superconductivity.
3. Even though niobium does not oxidize readily under ambient conditions, thin films a few tenths of a micron thick must be protected from the atmosphere. Otherwise they may lose their superconductive properties after several weeks' exposure.
4. The gas bearing apparatus must be modified to ensure random rotation of the supported sphere in order to provide a uniform coating.
5. Patterns for optical readout can be placed on the niobium by photoresist techniques and anodization of the niobium. These patterns can

be produced in any spectral color, are very resistant to abrasion, are unaffected by thermal cycling, and can be laid down with an accuracy of ± 0.002 in.

VI. RECOMMENDATIONS

As a result of this program the following recommendations are made:

1. Additional coated spheres should be produced, even though coated only near the equator, for evaluation of the superconducting properties, especially the Meissner effect and the critical magnetic field. Thicker coatings and both Lucalox and quartz substrates should be employed.
2. Chemical analysis of the coatings should be carried out to obtain information of possible pertinence to the superconducting behavior.
3. Vapor deposition equipment should be modified to permit better visibility of the sphere, more efficient utilization of the NbCl_5 , and a random rotation of the sphere.
4. If satisfactory superconducting properties are achieved, efforts must be made to measure the uniformity of the coating and the mechanical properties of the spheres. Additional work needs to be carried out on the grinding and polishing of Lucalox if this substrate continues to be of interest.

VII. TABLES AND FIGURES

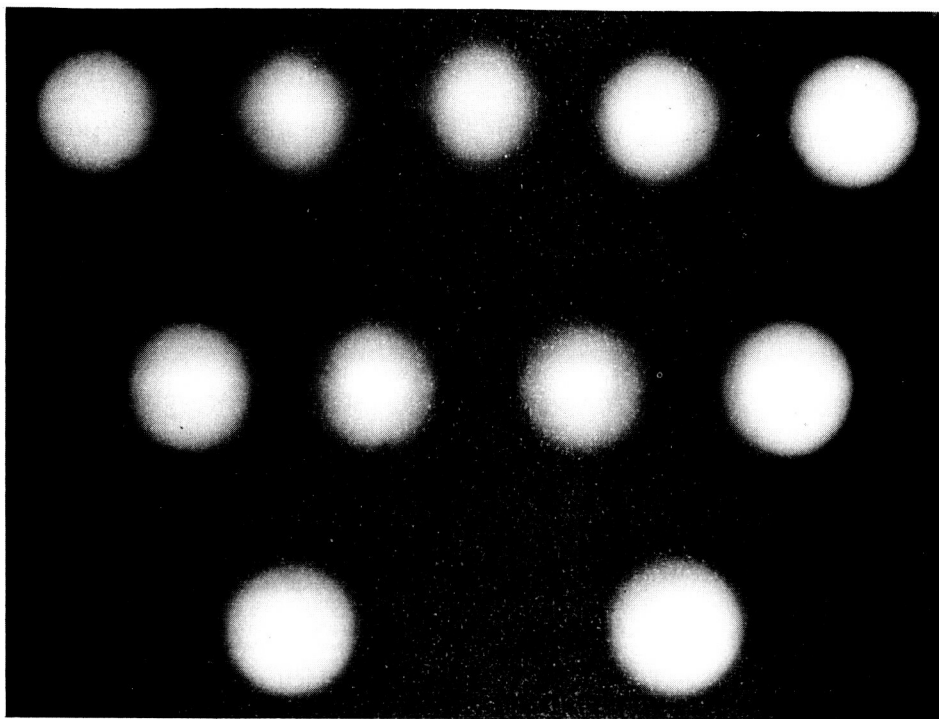
TABLE 1
Analysis of NbCl₅ Powder*

Metal	PPM	Method
Ta	146	Spec
B	<2	Spec
Cd	<1	Spec
Co	<3	Spec
Mo	150	Spec
P	30	Spec
Zr	Not Detectable	Spec
W	<200	Wet

* NbCl₅ obtained from the Stauffer Chemical Corporation, New York, N. Y., in 10 lb. lots.

TABLE 2
Typical Coating Run

Time (minutes from start)	Operation
0	<p>Sphere in bearing, chamber sealed, system evacuated and flushed with argon</p> <p>Chamber pressure regulated at 15 inches Hg with argon flowing at 25 cfh</p> <p>Strike plasma arc at 50 amps and increase slowly to 200 amps during next 60 minutes</p>
40	<p>Continue heating sphere</p> <p>Turn on heating mantles of NbCl_5 still and transfer line, and heat to $460\text{--}500^\circ\text{F}$</p>
60	<p>Start flow of hydrogen to still at 5 cfh</p> <p>(Plating has started with sphere at about 1000°C and NbCl_5 vapor being carried to chamber)</p> <p>Continue plating for about 20 minutes, depending upon quantity of NbCl_5 in still</p>
80	<p>Shut down still, transfer line and carrier gas</p> <p>Cool mantle of still with air</p> <p>Decrease plasma arc from 200 amps slowly to 50 amps in 30 minutes</p>
110	<p>Turn off plasma arc, but continue flow of gas for bearing</p> <p>Cool sphere 30 minutes longer</p>
140	<p>Turn off argon and water. Remove sphere.</p>



RF-8079

Fig. 1 - Radiograph of Lucalox spheres. (By X-ray 75 KV-5 ma-
8 min. Type M, Kodak Industrial X-ray film).

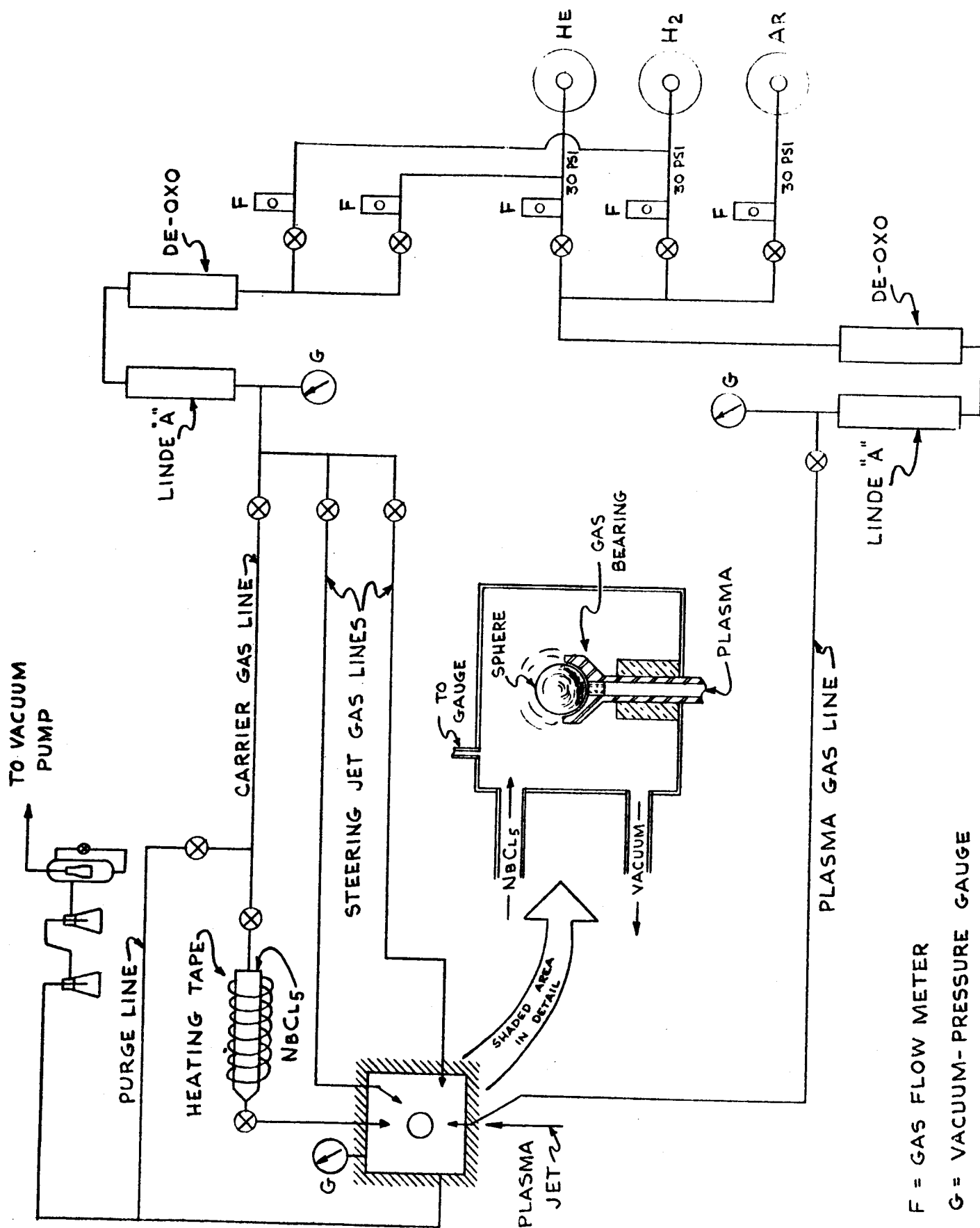
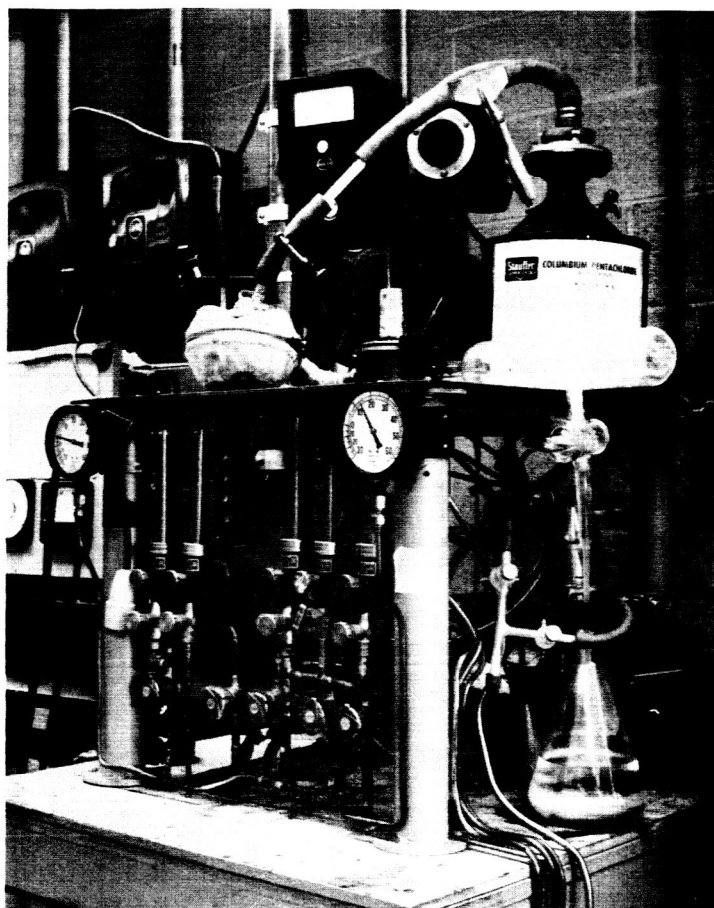


Fig. 2 - Schematic of deposition apparatus. Drawing No. RA-2061.



RF-8424

Fig. 3 - Photograph of deposition apparatus.

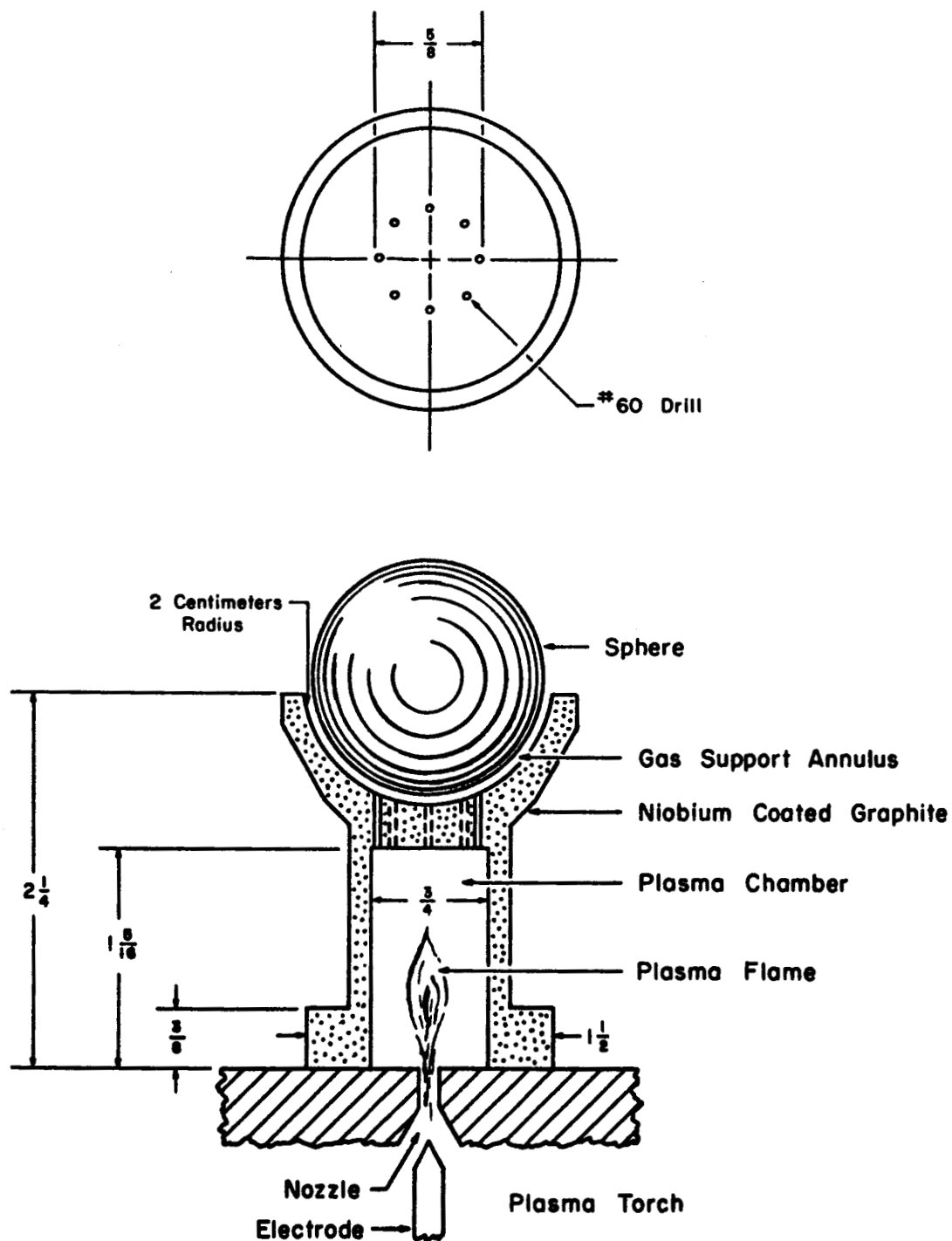


Fig. 4 - Hot-gas bearing and structure to distribute hot gas and decouple flame from sphere. Structure machined from graphite and coated with niobium vapor deposited from NbCl_5 .

Drawing No. RA-2158

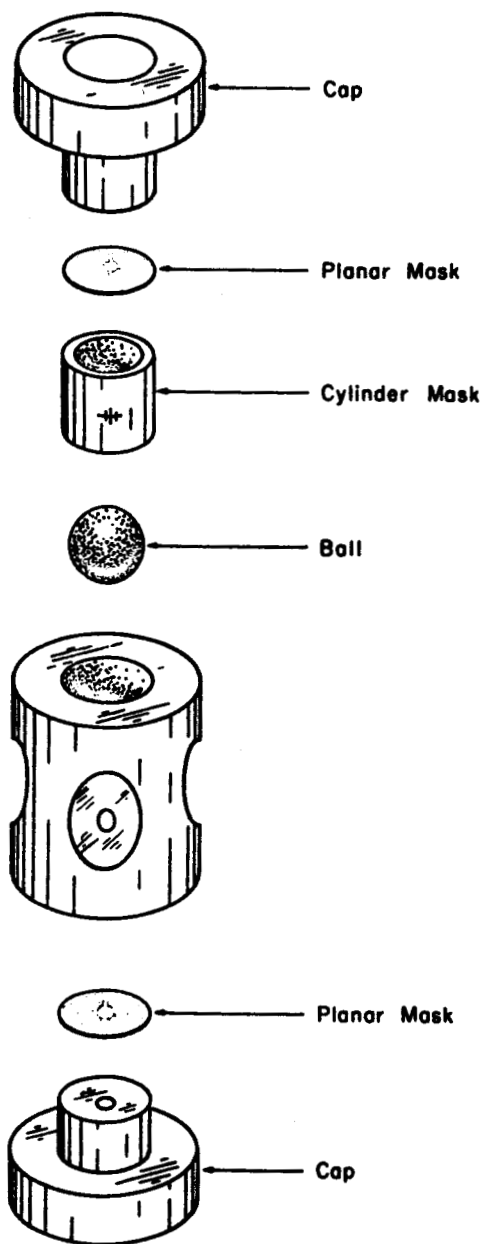


Fig. 5 - Exploded view of ball exposure cage with masks. Drawing No. RA-2265

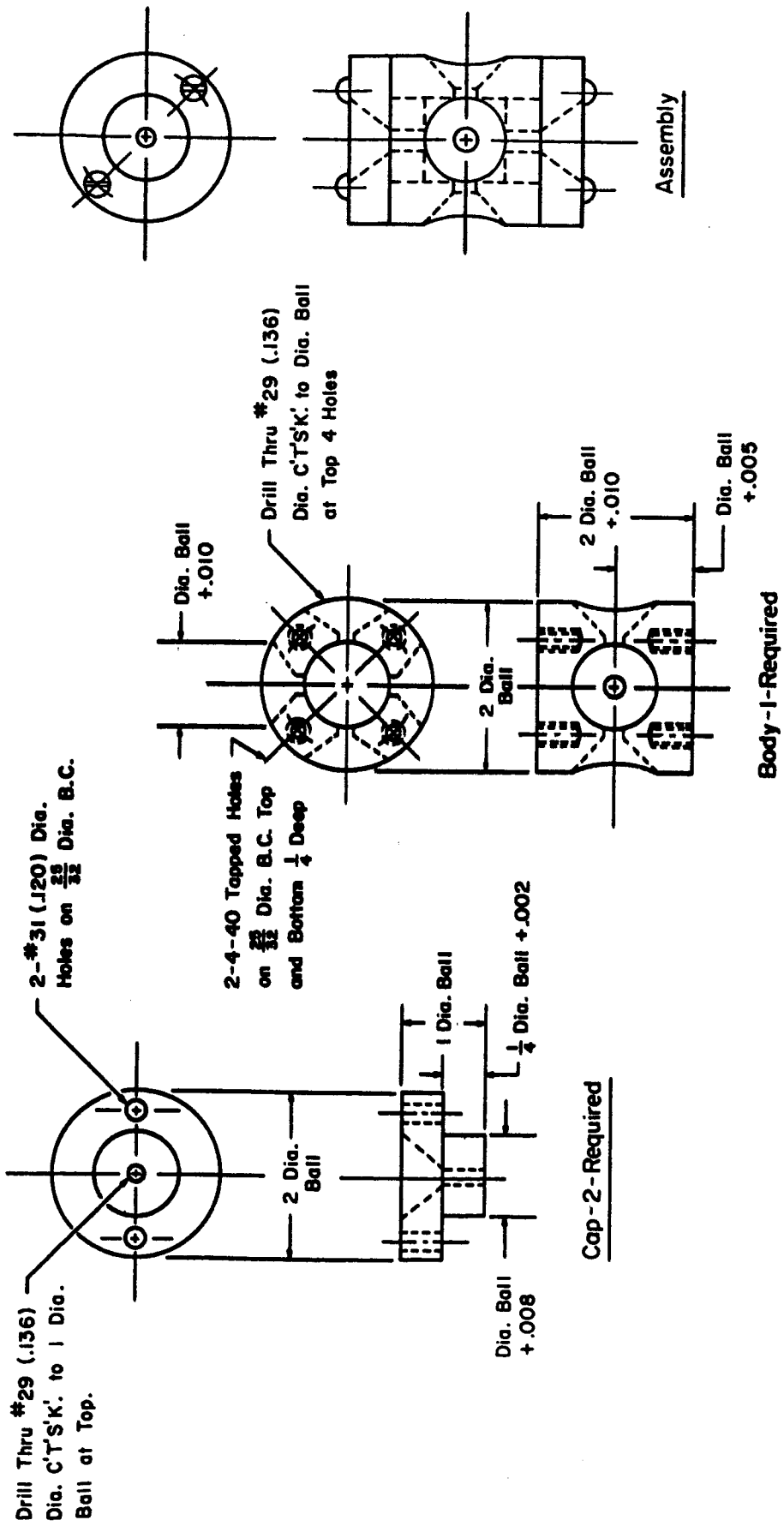


Fig. 6 - Ball exposure cage. Drawing No. RA-2264

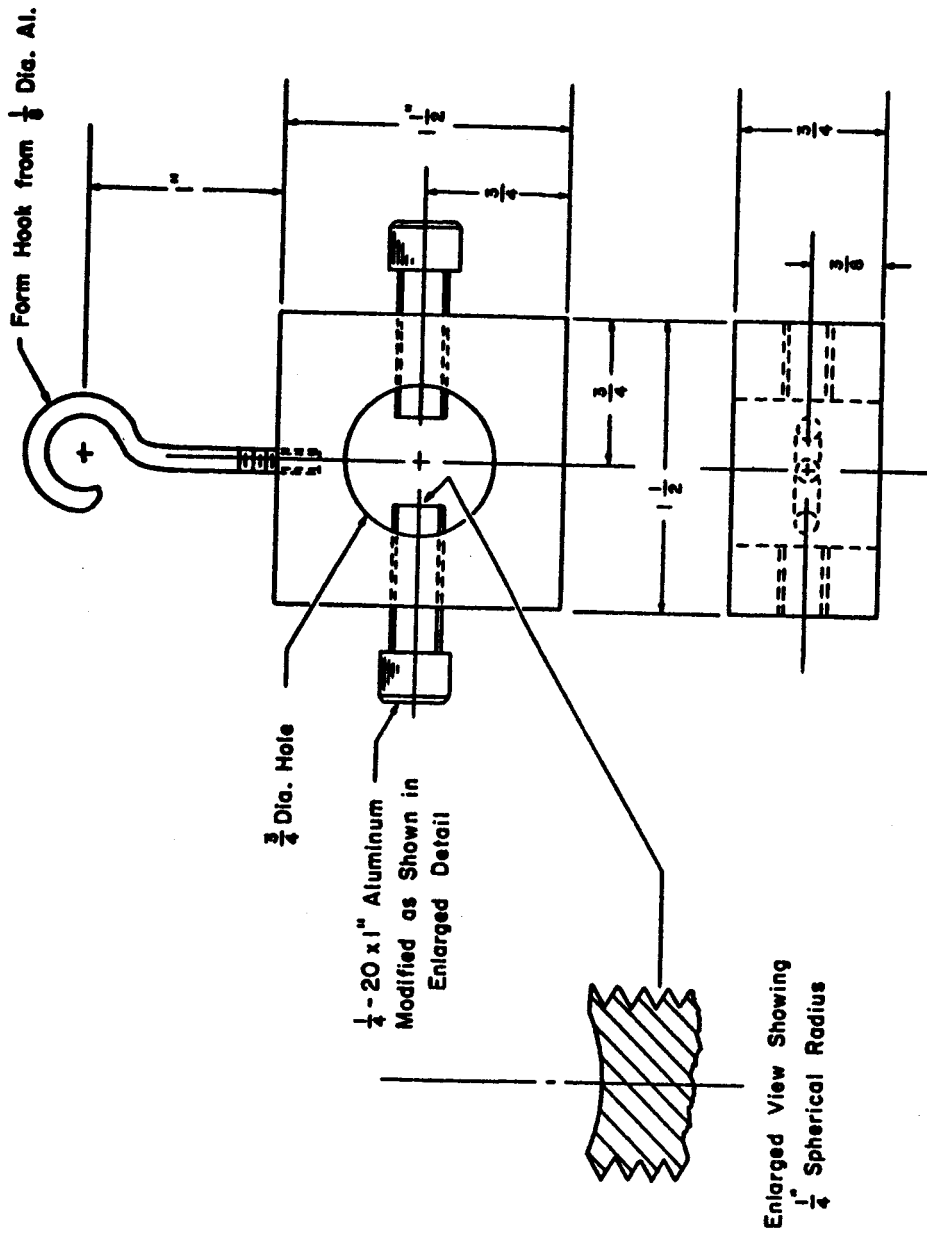


Fig. 7 - Anode fixture. Drawing No. RA-2263

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IX. ACKNOWLEDGMENTS

The authors wish to thank Messrs. A. Dodge and H. Leahy for their assistance in the construction and operation of the equipment. Thanks are especially extended to Mr. A. Lumbert for suggestions on equipment design and for help with this report.

APPENDIX A - SPECIFICATIONS

1. SCOPE

1.1 This document specifies the design requirements to develop and fabricate a spherical rotor for a cryogenic gyroscope.

2. APPLICABLE DOCUMENTS

None

3. REQUIREMENTS

3.1 Physical Properties

3.1.1 Size - The physical size shall be 4.00 ± 0.01 cm (centimeters) in diameter.

3.1.2 Weight - The completed rotor shall weigh not less than 20 grams nor more than 150 grams.

3.1.3 Sphericity -

(a) Variation of radius of surface with respect to geometrical center shall be less than 1×10^{-5} cm.

(b) Mass unbalance shall be less than (mass of rotor $\times 10^{-5}$ cm).

(c) The rotor may have a preferred axis, however, the variation in the moment of inertia in the plane perpendicular to the preferred axis and containing the center of mass must be less than (mass of rotor $\times 10^{-10}$ cm²).

3.2 Mechanical Properties

3.2.1 Stability - The sphericity shall meet the requirements of 3.1.3 at all temperatures between 4°K and 300°K, i.e., the rotor must have isotropic thermal expansion.

3.2.2 Spinning - When spun at 200 cps at 4.2°K the oblateness of the sphere must be such that the difference in length of the principle axes is less than 5×10^{-5} cm.

3.3 Rotor Surface

3.3.1 Superconductivity - The surface shall be superconducting.

(a) When superconducting, the penetration of the magnetic field into the rotor shall be uniform; the penetration shall not exceed 5×10^{-6} cm.

(b) The transition temperature shall exceed 7.0°K .

(c) The critical field at 4.2°K shall exceed 1900 gauss.

3.3.2 Hardness - The surface shall have a Brinell hardness greater than 100.

3.3.3 Corrosion - The surface must be resistant to atmospheric sources of corrosion.

3.3.4 Optical Pattern - A technique, for applying an optical pattern of the surface of the sphere, must be demonstrated. The optical pattern will permit photocell readout of the sphere orientation without affecting the magnetic properties or the mass balance.

APPENDIX B - BACKGROUND

I. SELECTION OF MATERIALS

A. Superconductor

Niobium (columbium) has been chosen on the basis of its satisfactory physical properties, the prior development work on coatings of niobium, and unsuitability or lack of data for alternate materials.

Alloys, although of high transition temperature and critical field, have been eliminated because of the physical-metallurgical problems associated with the intermediate state in the superconducting region. It would be tangential to the present objective to investigate the technology involved in achieving the high degree of homogeneity and uniformity needed here. In addition, many of the basic problems that exist for pure niobium would still be present.

Compounds are in much the same category as the alloys regarding uniformity and superconducting characteristics. Nitriding or carbiding the surface of the rotor (coated with molybdenum, tantalum, zirconium, or niobium) appears to be a rather undeveloped area of technique with serious questions regarding uniformity, roughness, stoichiometry, and cohesion.

Among the pure metals, only niobium fulfills the specifications. It has a transition temperature of 8°K , a critical field strength of 1900 oersteds at 4.2°K , and adequate atmospheric corrosion resistance (detectable reaction of niobium with air begins at about 180°C for an exposure of 20 hours.)

The surface will probably have a Brinell hardness greater than 100. Dead-soft niobium has a hardness about 85, while niobium sheet cold rolled to 87.5% reduction and annealed at 1000°C has a hardness of 100. Vapor deposited niobium coatings (about 0.0005-inch thick) have hardnesses about 150.

In addition to these favorable properties, there exists a sufficiently developed technology for the fabrication of ductile, adherent

niobium coatings to indicate a high probability of success in producing niobium coated rotors.

B. Substrate

The choice of substrate material is dictated by the mechanical property specifications and by compatibility with the niobium coating. The substrate provides the necessary rigidity and isotropy of the rotor and must provide an adherent base for the coating.

A wide range of materials might be suitable if the rotor were hollow. However, there are great difficulties in the fabrication, assembly and balancing of hollow rotors unless very unusual (and undeveloped) techniques are used. Therefore, to achieve a minimum cost and relatively high probability of success, solid rotors have been selected for this program.

Materials that have been considered for solid rotors include beryllium (single crystal and polycrystalline), sapphire, aluminum oxide, fused quartz, Pyroceram, sodium-silicate glass, and graphite. Graphite has been rejected because of roughness and porosity problems. The remainder of the materials are listed in Table B-1 with their linear thermal expansion coefficients and with computed strains in a niobium coating. Sapphire and single crystal beryllium were eliminated on the basis of their expansion anisotropies. Polycrystalline beryllium and aluminum oxide are possible rotor materials provided they are of high density and fine grained, randomly oriented structure. The silicate glass is suitable only for coating processes below 500°C. Pyroceram is reputed to be susceptible to dimensional changes if mechanically shocked. To avoid plastic deformation of the niobium, the strain should be well below 0.001. On these bases, only Al_2O_3 is suitable for high temperature coating processes, whereas all the materials listed in Table B-1 are suitable for low temperature processes. At this date, however, it is not certain whether the indicated strains will be detrimental or what fraction of the resultant working will be removed by the annealing during slow cooling from the high temperatures.

Further selection among these materials was made by consideration

of the oblateness specification. Casual discussion of the calculation of oblateness with Professors Eric Reissner and Jacob Den Hartog of the Massachusetts Institute of Technology indicated that, although no solution of this problem has been set forth to their knowledge, a solution accurate to perhaps 10% could be found in a short time using standard membrane theory. For the purposes of this proposal, however, an arbitrary distribution of strain between equator and poles was assumed and simple hoop stress calculations were used for the equatorial stress. Table B-2 is a tabulation of the results of calculations under these assumptions. Pyroceram has been eliminated because of its instability and sodium-silicate glass because of its low rigidity, low softening point and the special manufacturing runs that would be needed for its procurement. The choice of substrate materials has, therefore, been narrowed down to beryllium, Al_2O_3 , and fused SiO_2 .

As discussed in the next section, a high temperature coating process is preferred, and in this case Al_2O_3 becomes the first choice. Fused, polycrystalline, high density Al_2O_3 is commercially available, and present technology for successfully depositing adherent pure niobium on a UO_2 substrate indicates that a similar process for Al_2O_3 may be used. Fused Al_2O_3 is available in the necessary sizes from the General Electric Co. under the brand name of Lucalox. It is a translucent, polycrystalline 99.9+ percent pure Al_2O_3 of 99.8% theoretical density. Microstructural examination of Lucalox shows it to be free of the porosity usually found in Al_2O_3 bodies. The in-line transmission of Lucalox is about 35% for a wavelength of 0.7 micron and 83% for a wavelength of 5.5 microns for a sample 0.5-mm thick (uncorrected for surface reflection losses). Sufficient transmission may be present to enable visual or photographic inspection of the Al_2O_3 sphere for internal flaws and density variations. A similar inspection can be made of optical quality fused SiO_2 spheres. Beryllium spheres can be radiographed for internal density variations. Optical inspections, however, are far more accurate than those relying on X-rays in spite of the very low absorption of beryllium. Isotropy of the substrate material can be demonstrated by X-ray diffraction analysis for preferred orientation.

An additional reason for the preference of Al_2O_3 is that a rotor made of this material will be close to the maximum allowable mass of 150 grams, actually about 134 grams. Since the permissible mass unbalance is proportional to the mass of the rotor (Appendix A, Section 3.1.3), this allows a maximum of absolute unbalance and therefore permits deviations in coating thickness greater than those with a lighter rotor.

II. SELECTION OF PROCESSES

A. Rotor Fabrication

Aside from speculative schemes, grinding presently seems the most straightforward method of achieving the necessary sphericity. Machine grinding and lapping followed by hand-working is used by most facilities and therefore the fabrication of the spheres is extremely slow and expensive. For example, the quotations we have received for spheres ground to a sphericity of less than 1×10^{-5} cm (3.9 microinches) indicate that about four months are required to deliver the first sphere and that from two to four weeks per sphere are needed thereafter. The costs are between \$18,000 and \$25,000 for six spheres.

However, we have not used the above expensive method of rotor fabrication, although the "ultimate" gyroscopes may be made in this way. Rather, we utilized an "off-the-shelf" process that is reputed to produce ceramic or glass spheres with sphericity to 10 microinches with a good probability of substantial yield of spheres with sphericity below 5 microinches. This purported accuracy is possible only by virtue of special, imported machinery. If the sphericity is less than 5 microinches, it will be uniformly so over each sphere and for all the spheres in that run. Sphericities less than 10 microinches can be measured at the National Bureau of Standards for a modest charge. We feel that, for the purposes to be served in this proposed development work, these spheres will be adequate for demonstrating the feasibility of nearly all the specifications; the spheres are inexpensive and readily available so that the demonstration of feasibility can be performed at relatively low cost and in a minimum time.

Should it be necessary to consider the use of beryllium spheres, or to make spherical a niobium coated sphere, the situation is not as favorable as outlined above. With a relatively ductile metal, sphericity accuracies near 100 microinches are realized with the special machinery. However, the hand-work procedures are also applicable to the metals at the same cost and delivery schedules.

B. Niobium Coating

The required penetration depth of the magnetic field is not to exceed 5×10^{-6} cm (Appendix A, Section 3.3.1a). A coating thickness at least 20 times this penetration depth should provide bulk properties, provided the coating otherwise has the true niobium metallic structure. Thus, a coating thickness between 1 and 10 microns will be attempted. The technique used in putting on the niobium coating should achieve: high niobium-purity, uniform thickness, excellent adherence to substrate, and a continuous structure of niobium metal. Three techniques stand out as possibilities: evaporation, sputtering, and vapor-deposition. We have selected the last as the prime choice, although sputtering offers intriguing untested possibilities especially for beryllium substrates. The following tabulation lists some of the advantages and disadvantages of the evaporation and vapor-deposition techniques:

Vapor-Deposition (from NbCl_5)

<u>Advantages</u>	<u>Disadvantages</u>
1. Easy to achieve uniformity; deposition not "line-of-sight".	1. May be difficult on beryllium; BeO and BeCl_2 problems.
2. Easy to suspend sphere; gas bearing possible.	2. Substrate at 700 to 1000°C; NbCl_5 at about 140°C.
3. Demonstrated feasibility on ceramic and metal; good adhesion and metallic structure.	3. Need to produce NbCl_5 in pure form.

Evaporation (from Nb filament)

- | | |
|-------------------------------|---|
| 1. No controlled atmospheres. | 1. Sphere suspension problems. |
| 2. Purity easy to achieve. | 2. Difficult geometrical control for uniformity. |
| 3. Substrate at 30 to 500°C. | 3. Good adhesion and continuous metallic structure may be difficult to achieve. |

Numerous investigators have ascertained the conditions necessary for successful, rapid vapor-deposition of niobium from NbCl_5 . These techniques are currently being used in Atomic Energy Commission programs to coat UO_2 with adherent, dense, non-porous niobium capable of being temperature cycled over the minimum range of 30 to 200°C without cracking.

Table B-1

Linear Thermal Expansion Coefficients and Niobium Strain for Various Substrate Materials

Substrate Material	Linear Thermal Expansion coeff. $\times 10^6$ ($^{\circ}\text{C}$) $^{-1}$ [Range in ($^{\circ}\text{C}$)]	Nb Strain (800 to -200°C)*	Nb Strain (0 to -200°C)*	Remarks
Al_2O_3	8.1 [25 - 900] 6.7 [25 - 300]	+0.00012	-0.00012	Fused, polycrystalline, high density
Sapphire	8.8 [20 - 800] 8.1 [20 - 800] 6.7 [20 - 50] 5.0 [20 - 50]			// to C-axis ⊥ to C-axis // to C-axis ⊥ to C-axis
SiO_2	0.53 [25 - 900] 0.26 [(-191) - 16]	-0.0069	-0.0011	Fused, optical quality
Be	17.2 [25 - 700] 8.1 [(-120) - 0] 18 [800]; 15 [400]; 9 [50] 22 [800]; 18 [400]; 12.5 [50]	+0.008	+0.00046	Random, polycrystalline Single crystal, // to C-axis Single crystal, ⊥ to C-axis
Pyroceram 9606	5.7 [25 - 300]	-0.0016	-0.0003	Susceptible to dimensional instability by mechanical shocking
Sodium-silicate glass	7.3 [0 - 250]	0	0	85% SiO_2 ; softens at 500°C
Nb	7.8 [18 - 900] 5.8 [(-212) - 0] 7.3 [18 - 300]			

*Assume niobium deposited at 800°C and the difference in expansion coefficients is zero below -200°C . Plus (+) strain indicates niobium is in compression; minus (-) strain indicates niobium is in tension.

Table B-2

Ranking of Various Substrate Materials for Their Resistance to Oblateness

Substrate Material	Density (ρ) g/cm ³	Young's Modulus (E) $\times 10^{-6}$ lb/in. ²	Rigidity Index* $(\frac{\rho}{E}) \times 10^6$	Hoop Stress** σ_h (psi)	$\frac{\sigma_1}{\sigma_h}$ ***	$\frac{\sigma_2}{\sigma_h}$ ***
Be	1.85	44	0.042	170	1.6	2.2
Al ₂ O ₃ (fused polycrystalline)	4.00	50+	0.080	370	0.85	1.12
Pyroceram	2.61	17.3	0.15	240	0.45	0.60
Sodium silicate glass	2.30	10.0	0.23	210	0.30	0.40
SiO ₂ (fused)	2.20	10.5	0.21	200	0.33	0.44

*Low values of this rigidity index favor low oblateness.

Equator-ring hoop stress. $\sigma_h = \frac{2}{\rho w R^2} = 91.5\rho$ (psi); where ρ = density in g/cm³, $w = 400\pi$ rad/sec, $R = 2$ cm.* σ_1 and σ_2 are stresses computed from oblateness specification assuming strain is distributed 1:1 and 1:2 polar:equatorial, respectively. $\sigma_1 = \frac{25}{4} E \times 10^{-6}$ psi, $\sigma_2 = \frac{25}{3} E \times 10^{-6}$ psi; where E = Young's Modulus in psi. σ/σ_h must be greater than unity to meet specifications.